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09/743623

TRANSMITTAL LETTER TO THE U.S. ELECTED OFFICE
(EO/US) - ENTRY INTO NATIONAL STAGE UNDER 35 USC 371

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International Application No.

15 July 1999

International Filing Date

15 July 1998

Priority Date Claimed

PROCESS AND APPARATUS FOR MANUFACTURING A GLASS INGOT
FROM SYNTHETIC SILICA

Title of Invention

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Box PCT
Assistant Commissioner for Patents
Washington, DC 20231
Attn: EO/US

Sir:

Applicant herewith submits to the United States Elected Office
(EO/US) the following items under 35 USC 371:

- (1) This express request to immediately begin national examination procedures (35 USC 371(f)).
- (2) A copy of the cover sheet for the published International Application along with a copy of the specification as filed: 22 pages, including 2 pages of claims, 4 sheets of drawings, and a copy of the 3 page International Search Report.
- (3) a copy of the 4 page Request form.
- (4) a first Preliminary Amendment for entry prior to calculation of the filing fees.

Express Mail No.

EK992703395US

- (5) our check in the amount of \$860.00, covering the basic national fee as set forth in 37 CFR 1.492(a)(5) and based on the first Preliminary Amendment.
- (6) A Second Preliminary Amendment.

Copies of the following miscellaneous items are also enclosed:

- (7) Copy of the 3 page Demand for International Preliminary Examination.
- (8) Copy of the 7 page Written Opinion.
- (9) Copy of the 6 page Response to the Written Opinion.
- (10) Copy of the 9 page International Preliminary Examination Report.
- (11) Copy of the 1 page Notification of the Recording of a Change.

The Combined Declaration and Power of Attorney form will be filed by the appropriate deadline under 37 CFR §1.495(c)(2) with the surcharge under 37 CFR §1.492(e).

Please charge any additional fees which may be required to effect entry into the National Phase and credit any overpayment to Deposit Account No. 08-3040.

Please direct all communications concerning this application to the undersigned.

Respectfully submitted,

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Attorneys for the Applicants

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09/743623

JC07 Rec'd PCT/PTO 12 JAN 2001

JYG143USA

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of) Group Art Unit:
)
Ian George Sayce et al) Examiner:
)
Appln. No.)
)
Filed: Herewith)
)
For: PROCESS AND APPARATUS FOR) January 12, 2001
MANUFACTURING A GLASS INGOT)
FROM SYNTHETIC SILICA)

Assistant Commissioner for Patents
Washington, DC 20231

SECOND PRELIMINARY AMENDMENT

Sir:

Please amend the above-identified patent application as follows.

In the Specification

Please enter the attached Abstract of the Disclosure on the attached page.

In the Claims

Kindly add new claims 11-23 as follows.

11. The method according to claim 1, wherein the shaping orifice is located at the lowest part of the mass in the refractory container and wherein said removing step comprises positively withdrawing the ingot from below.

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12. The method according to claim 11, further comprising adding synthetic silica to the mass at a rate substantially similar to that at which the ingot is withdrawn.

13. The method according to claim 1, further comprising the steps of maintaining the melt in a molten state by heating with one or more burners prior to said removing step, wherein in said method at least one burner is a synthesis burner, and the silica is deposited from such synthesis burner(s) in such a manner that synthetic vitreous silica can be deposited at a rate substantially similar to that at which silica is withdrawn as ingot through the shaping orifice.

14. The method according to claim 1, further comprising depositing synthetic silica into the refractory container from a synthesis burner.

15. The method according to claim 13, wherein the synthesis burner heats the surface of the melt so that the deposited silica sinters directly to glass.

16. The method according to claim 14, wherein the synthesis burner heats the surface of the melt so that the deposited silica sinters directly to glass.

17. A furnace for the manufacture of synthetic vitreous silica ingot, the furnace comprising: a furnace enclosure housing a refractory container, the container being adapted to hold a melt of synthetic vitreous silica; a die disposed within a wall or base of the container, the die including an orifice through which the glass ingot is extruded; moveable support means downstream of the orifice, adapted to support and facilitate withdrawal of the ingot; and one or more burners adapted to maintain the silica at or above its sintering temperature, wherein at least one burner is a synthesis burner.

18. The furnace according to claim 17, wherein said burner comprises associated means for the supply of silica precursor and combustion gases and is adapted to deposit synthetic vitreous silica by vapour deposition onto the surface of the melt, wherein said furnace permits continuous withdrawal of silica as ingot at a rate substantially similar to that at which silica is deposited by said synthesis burner.

19. The furnace according to claim 17, wherein the moveable support means comprises an arrangement of moveable clamps.

20. The furnace according to claim 19, wherein the refractory container with its die, the ingot and the arrangement of clamps can be rotated synchronously to provide a deposited glass of improved homogeneity.

21. The furnace according to claim 19, wherein the refractory container with its die, the ingot and the arrangement of clamps can be moved to and fro horizontally to permit spreading of the pattern of deposited glass from the burner.

22. The furnace according to claim 19, wherein the refractory container with its die, the ingot and the arrangement of clamps can be moved in orthogonally disposed x- and y- directions, to permit spreading of the pattern of deposited glass from the one or more burners.

23. The furnace according to claim 19, wherein spreading of the pattern of deposited silica is achieved by movement of the one or more burners and/or of the refractory container.

REMARKS

Upon entry of this second preliminary amendment the pending claims are Claims 1 and 11-23. New claims 11-23 are supported throughout the specification and by original claims 2-10, respectively, and have eliminated multiple dependencies. No new matter is added by this preliminary amendment.

The attached Abstract of the Disclosure is supported throughout the specification.

Applicants respectfully request consideration of the pending claims.

The Director of the U. S. Patent and Trademark Office is hereby authorized to charge any deficiency in any fees due with the filing of this paper or credit any overpayment in any fees to our Deposit Account No. 08-3040.

Respectfully submitted,

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ABSTRACT OF THE DISCLOSURE

A method and apparatus for the manufacture of synthetic vitreous silica ingots involves the production of a melt of synthetic vitreous silica in a crucible (35) within a refractory furnace (31), and the continuous withdrawal of an ingot (43) through an
5 orifice (40) in the wall of the crucible. The silica may be deposited in the crucible by a synthesis burner (33), which may also serve to maintain the silica above its sintering temperature. The emerging ingot is supported by an arrangement of moveable clamps (44).

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For: PROCESS AND APPARATUS FOR) January 12, 2001
MANUFACTURING A GLASS INGOT)
FROM SYNTHETIC SILICA)

Assistant Commissioner for Patents
Washington, DC 20231

PRELIMINARY AMENDMENT

Sir:

Please amend the above-identified patent application as follows.

In the Claims

Cancel claims 2-10 without prejudice.

REMARKS

After entry of this preliminary amendment, the pending claim is claim 1.
Claims 2-10 are canceled. No new matter is introduced by this preliminary amendment.

Applicants respectfully request that this preliminary amendment be entered prior to calculating the filing fees.

Express Mail No. EK99270339545

The Director of the U. S. Patent and Trademark Office is hereby authorized to charge any deficiency in any fees due with the filing of this paper or credit any overpayment in any fees to Deposit Account No. 08-3040.

Respectfully submitted,

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4. PRTJ

09/743623

JG07 REC'D PCT/PTO 12 JAN 2001

PROCESS AND APPARATUS FOR MANUFACTURING A GLASS INGOT FROM SYNTHETIC SILICA

Fused quartz glasses, typically made by fusion of refined natural quartz crystal powders, are widely used in the optical, optical fibre and semiconductor industries for their optical properties, chemical resistance, thermal stability or other properties.

In some critical applications, the impurities or inhomogeneities resulting from the use of a natural crystal feedstock may be unacceptable, and then it becomes necessary to substitute a synthetically produced material. Sometimes this may be a synthetically produced powder, but more generally the highest grade vitreous silica products are made by vapour deposition. Thus a vaporised precursor compound of silicon is fed to a synthesis flame where it is oxidised or hydrolysed to form a stream of silica fume or a flow of micro-particles of silica which is caused to deposit either as a porous silica soot body, which may be dehydrated or doped by heating in a suitable atmosphere, and then sintered to pore-free glass, or alternatively by deposition at such a temperature that the silica deposit sinters directly to a transparent glass.

The latter process, often called the direct deposition process, yields glass of relatively high OH (hydroxyl content), typically 800-1200ppM by weight, but this is acceptable for many applications, for optical components such as prisms, lenses etc., for larger articles such as windows of furnaces or spacecraft etc., for mirrors, and for the manufacture of photomasks, i.e., the plates which carry the images to be imprinted by a photolithographic process on a silicon wafer, during the manufacture of microcircuits.

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The direct deposition process may be operated in either of two modes. In the first of these, shown in Figure 1 of the accompanying drawings, a burner 11 provides a synthesis flame 12, typically an oxy-hydrogen flame, and is fed, via a central duct 11a with a stream of precursor material. The precursor material can be one or more gaseous chlorosilanes, (e.g. silicon tetrachloride), but more recently chlorine-free precursors have been finding favour. The silicon compound is oxidised or hydrolysed to form a stream of silica fume or a flow of micro-particles of silica which is directed on a substrate forming the domed end 13a of a rotating cylindrical ingot 13, supported within a furnace structure 14. A substantial proportion of the silica generated in the flame deposits on the substrate which is slowly withdrawn (in direction Z) from the furnace, preferably maintaining a substantially constant burner-to-substrate distance. The silica is deposited on the substrate at such a temperature that it sinters directly to transparent, pore-free glass. The ingot may be rotating about a horizontal, vertical or other axis, and may be subjected to oscillatory movement on either or both axes (X and/or Y) perpendicular to that of rotation to spread the thermal load on the ingot end 13a, and thus to increase the homogeneity of the glass deposited or to control the cross-sectional shape of the ingot.

A second geometrical arrangement used for collecting glass by direct deposition is shown in Figure 2 of the accompanying drawings. This employs a rotating shallow refractory crucible 21, typically lined with zircon or zirconia-based refractory bricks, mounted on a turntable 22. The bottom of the shallow crucible is typically lined initially with a layer of high purity quartz or quartz glass powder 23, or alternatively crushed synthetic vitreous silica glass for maximum purity. Over this crucible is mounted a refractory roof 24 which carries one or more synthesis burners 25. The crucible may be between 1 and 2 metres in diameter and under these circumstances a significant number of burners may be employed. These serve both to heat the crucible to a

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temperature above the melting point of silica, and also as generators of synthesis flames 26, each of which deposits a stream of silica fume or soot on the surface of a molten glass pool 27 which is generated in the crucible. After an appropriate thickness of glass has been so generated, the crucible is allowed to cool, the refractory walls are removed, and the disk-shaped ingot of glass is taken away to be cut, machined or otherwise formed into the required shape.

The process of Figure 1 can be used to generate a cylindrical (e.g. circular cylindrical) ingot. This may be of a size suitable for conversion into cylindrical sections if required, e.g., for lenses or mirror blanks, or may be converted by further thermal processes to rod or tube products. However for some applications a cylindrical shape may be an unsuitable starting material. Thus for certain applications where a series of square or rectangular products is required, for example for photomask substrates, these are either machined from an oversize ingot, with evident wastage, or alternatively the cylindrical ingot is re-shaped, for example by heating to softening temperature within a graphite mould of appropriate internal dimensions and, by slumping under its own weight, or by application of pressure, to force the softened silica to take up the shape of the mould. After cooling, the re-shaped ingot may be cut into slices of the desired dimensions. This secondary operation is costly and results in material losses.

Where such shapes are to be cut from one of the large disk-shaped ingots generated by the crucible process of Figure 2, this involves extensive cutting operations, and again much wastage. It may also be necessary to reject material of unsatisfactory quality, due, for example, to contamination from the refractories of the furnace roof, or from the crucible itself.

Thus for certain shapes of product, notably those of square cross-section, neither of the two major manufacturing

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methods yields an ingot which may be used directly, and with high materials efficiency. Furthermore neither permits continuous operation as would be desirable for more economical operation, since both are essentially batch processes.

5 There is thus a requirement for a direct deposition process for synthetic vitreous silica glass, which can be operated continuously and which will generate an ingot of predetermined cross-sectional dimensions, i.e., round, square, rectangular, or other.

10 This invention seeks to meet that requirement by providing an improved method of forming a shaped body of synthetic vitreous silica glass and an improved furnace for the manufacture of such a shaped body.

According to one aspect of the invention a method of
15 forming a shaped body of synthetic vitreous silica glass includes the steps of generating a mass of molten synthetic silica in a refractory container, part of the boundary of which defines a shaping orifice, and removing the generated synthetic silica from the container through the orifice as a
20 shaped ingot.

The refractory container (e.g. a crucible) is desirably contained within a refractory furnace enclosure. The silica within the container may be kept above sintering temperature by one or more burners, which may conveniently be supported
25 by the roof of the furnace enclosure so that the flame of the or each burner is directly downwardly towards the crucible. Preferably, the synthetic silica is produced by vapour deposition, in which case at least one of the burners should be a synthesis burner. Alternatively, pre-synthesised silica
30 may be supplied to the crucible, for example in the form of powder, crystal or amorphous grain.

Conveniently the shaping orifice is located at the lowest part of the mass in the crucible and the removal involves

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positively withdrawing the ingot from below, preferably at a rate substantially similar to that at which synthetic silica is being added to the mass.

Preferably, the burner(s) serve(s) both to generate the
5 synthetic silica in particulate form and to heat the melt so that the silica sinters directly to glass in the mass. Optionally additional heat may be imparted by further heating means.

According to a further aspect, the invention provides a
10 furnace for the manufacture of a synthetic vitreous silica ingot, the furnace comprising: a furnace enclosure housing a refractory container, the refractory container being adapted to hold a melt of synthetic vitreous silica; one or more burners extending into the furnace enclosure and adapted in
15 operation to maintain vitreous silica within said container at or above its sintering temperature; a die disposed within a wall of said container, the die including an orifice through which the glass ingot is extruded; and an arrangement of moveable clamps downstream of the orifice, adapted to support
20 the extruded ingot.

Preferably, at least one burner is a synthesis burner, adapted both to deposit synthetic vitreous silica into the refractory container (e.g. crucible) and to assist in maintaining such silica above its sintering temperature. In
25 such an arrangement, the apparatus also includes means to feed oxygen, fuel and silicon-containing precursor material to the or each synthesis burner.

Optionally, the crucible with its die, the ingot and the arrangement of clamps may be rotated synchronously to provide
30 a deposited glass of improved homogeneity.

Again optionally, the crucible with its die, the ingot and the arrangement of clamps may be moved to and fro horizontally in an x-direction, or alternatively in

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orthogonally disposed x- and y- directions, to permit spreading of the pattern of deposited glass from the one or more burners.

Alternatively a spreading of the pattern of deposited silica can be achieved by like movement of the burner array and the furnace enclosure.

The invention will now be more fully described, by way of example, with reference to Figures 3 and 4 of the accompanying drawings.

10 In one embodiment of the invention, as illustrated in Figure 3, the apparatus comprises a furnace enclosure 31 lined with refractory brick and supporting a roof 32 also made of suitable refractory material. One or more burners 33, project through this roof. These burners may be made from metal or
15 quartz glass, and are fed with a fuel gas (e.g., hydrogen, and/or natural gas etc), with oxygen, and with the vapour of an appropriate compound of silicon, which on oxidation and/or hydrolysis yields a stream of micro-particles of silica fume, which stream is directed at the surface of a mass 34 of glass
20 melt contained in a refractory container or crucible 35.

The precursor material can be silicon tetrachloride or other halosilane, in which case the product gases contain the noxious and corrosive by-products, hydrochloric acid and chlorine. These must be handled with care, and scrubbed with
25 appropriate gas-cleaning equipment before release to atmosphere. Alternatively, if the precursor material is a chlorine-free silicon compound such as a siloxane or alkoxysilane, then the product gases contain only carbon dioxide, water vapour, and uncollected silica fume, and
30 effluent treatment is greatly simplified.

There exists a wide range of potential siloxanes which may be used in the burners 33, but preferred siloxanes are the

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polymethylsiloxanes, including the linear polymethylsiloxane hexamethyldisiloxane, and the cyclic polymethylsiloxanes octamethylcyclotetrasiloxane (OMCTS) and decamethylcyclopentasiloxane (DMCPS). Of the variety of 5 alkoxy silanes which may alternatively be used, one of the preferred precursors is methyltrimethoxysilane (MTMS).

As an alternative, synthetic vitreous silica (for example produced from any of the abovementioned precursor materials) may be supplied directly to the crucible in the form of 10 powder, crystal or amorphous grain, rather than being deposited there by a synthesis burner.

The product gases are led from the furnace via exhaust ducts 36, 37, their flow being controlled by means of valves 38, 39.

15 As noted, the refractory enclosure 35 serves as a crucible to contain the glass melt, and in the base of this crucible is an orifice 40 which may be defined by a die brick, or other die assembly 40a, constructed to form an exit which serves as a continuous casting nozzle through which molten 20 glass is progressively drawn in the course of the process. This die brick or die assembly may be made of a refractory material similar to that used to line the vessel 35, or may be made from another refractory material selected for improved erosion resistance, or may even be made from a refractory 25 metal, optionally protected by a ceramic coating (e.g., molybdenum, coated with molybdenum disilicide). If a refractory metal die orifice is used, it is preferable to surround any exposed metal (e.g., the underside of the lip of the die) with a reducing gas, such as a hydrogen-nitrogen gas 30 mixture.

Crucible 35 is supported, via appropriate insulating bricks 41, on a fixed base plate 42.

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Beneath the orifice 40 depends a glass ingot 43, supported by a series of moveable clamps 44, which are designed during normal operation to move progressively downwards encouraging withdrawal of melt from crucible 35 at a selected rate, (e.g. corresponding to the rate at which synthetic silica glass is deposited from above by the array of burners 34). At intervals, each one of these clamps may be caused to release its grip from the ingot, and independently be driven upwards to the upper limit of its motion, before being adjusted once again to grip the descending ingot. In this way the clamps cycle up and slowly down, and the glass ingot is maintained under steady motion downwards. By ensuring that at all times the ingot is gripped by at least two clamps, the ingot is maintained entirely straight.

At intervals, a length of the glass ingot 43 may be cut off from its lower end, e.g., at point 45 accessible from mid-floor level 46, and then lowered to ground floor level 47, where it is released and removed for further processing.

By selecting the dimensions of the orifice 40 it is possible in this way continuously to form synthetic vitreous silica ingot of predetermined cross-section and dimensions. Thus it is possible to make cylindrical ingot, or ingot of square or rectangular cross-section, and even to extrude a rectangle of high aspect ratio, i.e., plate. While for simplicity the base of crucible 35 is shown as substantially flat in Figure 3, for some applications it may be preferable to have an alternative shape, e.g., frusto-conical, to facilitate the flow of the glass to the orifice 40, to ensure an appropriate temperature distribution at the walls of the vessel, and to minimise devitrification at the walls, or around the orifice, which might otherwise affect the dimensions of the extruded ingot.

The process depicted in Figure 3 can be commenced as follows. The crucible 35 is assembled on the base plate 41,

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and a die assembly 40a is inserted. A previously manufactured ingot is raised via the clamps 44 into the orifice 40. This ingot may be machined if necessary to be an exact fit in the die orifice, and serves as a "bait-piece". The base of the crucible is covered with previously manufactured synthetic vitreous silica (e.g. in the form of lumps of glass). The furnace is brought to temperature by heating with the burner array, causing the initial furnace fill to melt and fuse to the upper end of the protruding bait-piece. Precursor material is then fed to the burners and, as deposition of glass proceeds, the melt level in the crucible 35 rises. When the desired melt depth is achieved, ingot retraction is commenced by starting the progressive downward motion of the clamps 42. Manufacture of ingot then continues as a steady process, with ingot withdrawal at least substantially matching glass deposition rate, and with ingot sections being cut off and removed at intervals as required (e.g. regular intervals).

The process depicted in Figure 3 is convenient for many applications requiring a glass ingot of high purity and controlled dimensions. However, as depicted, the crucible 35 is stationary, and it is evident that each burner 33 is directed at a fixed area of the surface of the melt. Glass deposited in this region thus has a slightly different hydroxyl level than that deposited elsewhere, in cooler regions. If a chlorine-containing precursor material is used, then the chlorine content of the glass will be higher in the region of impact of each synthesis flame. These effects can result in minor inhomogeneities in the chemical properties and also in the refractive index of the glass ingot. For this reason it may be desirable to rotate the crucible 35 and ingot 43 in the course of the process and this is achieved via a development of the process, shown in Figure 4.

In this case the apparatus comprises a furnace enclosure 51, lined with refractory brick, and supporting a roof 52, also made of suitable refractory material. One or more burners 53 project through this roof. These burners may be

- 10 -

made of metal or quartz glass, and are fed with fuel gases, oxygen, and precursor vapour as described above.

The product gases are led from the furnace via exhaust ducts 56, 57, their flow being controlled by means of valves 58, 59.

Again the product glass is collected in a refractory vessel or crucible assembly 55 in the base of which is disposed a shaping orifice 60, defined by a refractory die 60a made of one or more bricks, or alternatively a refractory metal plate as described above. The crucible assembly 55 is again supported on insulating bricks 61 and on a base plate 62, but in this case base plate 62 comprises a turntable, which is maintained at constant height, but is capable of rotation about a vertical axis.

15 Beneath the shaping orifice 60, depends a glass ingot 63, supported by a series of rotating chucks 64, which are designed to rotate synchronously with the crucible assembly 55, and turntable 62, but are also capable of progressive advance in a downward direction, all chucks moving at
20 identical speed, thus permitting withdrawal of melt from the crucible assembly 55 at a chosen rate (preferably a constant rate, corresponding to the rate at which synthetic silica glass is deposited by the array of burners above).

Again, as previously, each one of these chucks 64 may be
25 caused to release its grip from the ingot 63, and can then be independently driven upwards to the upper limit of its motion, before being adjusted once again to grip the descending ingot. In this way the chucks 64 cycle up and slowly down, while rotating at constant speed, and the glass ingot is maintained
30 with constant rotation and steady motion downwards. By using twin-jawed chucks 64 it is possible to ensure that the ingot 65 is maintained entirely straight.

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At intervals, a length of glass ingot may be cut off, e.g., at point 65, at mid-floor level 66, and then lowered to ground floor level 67, where it is released and removed for further processing.

5 Again, by selecting the dimensions of the shaping orifice 60, it is possible continuously to form a synthetic vitreous silica ingot of predetermined cross-section and dimensions. Thus it is possible to make a circular cylindrical ingot, or an ingot of square or rectangular cross-section, and even to
10 extrude an ingot whose cross-section is a rectangle of high aspect ratio, i.e., plate. While for simplicity the base of the crucible assembly 55 is shown as substantially flat in Figure 4, for some applications it may be preferable to have an alternative shape, e.g., frusto-conical, to facilitate the
15 flow the glass to the shaping orifice 60, to ensure an appropriate temperature distribution at the walls of the vessel, and to minimise devitrification at the walls, or around the orifice 60, which might otherwise affect the dimensions of the extruded ingot.

20

Because of the rotation of the crucible assembly 55, the burners 53 are depositing on a continuously moving surface, and this avoids overheating of any localised area on the glass surface, enhances the deposition efficiency and enables
25 a substantial increase in homogeneity of the glass deposited in the crucible assembly to be achieved. Additionally, the progressive motion of the glass through the crucible assembly to the orifice 60, permits mixing and diffusion processes which further enhance the homogeneity of the glass product.

30 An installation comprising the turntable 62, and the moving chucks 64, can be considered to be a large vertically-oriented lathe, of which the bed comprises a tower assembly 68. For the ultimate in homogeneity, it is possible to cause the supporting tower 68 to move horizontally to and fro in an
35 x- direction, or even in both x- and y- directions these directions being mutually at right angles and at right angles

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to the direction Z of ingot withdrawal, but this is unnecessary for most applications of the glass ingot product. Alternatively, it is possible in principle to cause the furnace roof/burner assembly to oscillate slowly in the x-
5 direction, and potentially in the x- and y- directions, to effect the same homogenisation process.

The choice of refractories is clearly important for successful operation of this type of process. In general high quality zircon refractories have proved adequate, but high
10 purity is necessary to minimise contamination, especially when using chlorine-free precursors. Greater erosion resistance is however achieved when using yttria stabilised zirconia refractories, the added expense of which is justified by the increased longevity of the furnace components, and the
15 efficiency of the process in enabling the manufacture on a continuous basis of an ingot of the required cross-section and dimensions.

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CLAIMS

1. A furnace for the manufacture of synthetic vitreous silica ingot, the furnace comprising: a furnace enclosure housing a refractory container, the container being adapted to hold a melt of synthetic vitreous silica; a die disposed within a wall or base of the container, the die including an orifice through which the glass ingot is extruded; moveable support means downstream of the orifice, adapted to support and facilitate withdrawal of the ingot; and one or more burners adapted to maintain the silica above its sintering temperature; characterised in that at least one burner is a synthesis burner, such burner(s) being provided with associated means for the supply of silica precursor and combustion gases and being adapted to deposit synthetic vitreous silica by vapour deposition on to the surface of the melt, the arrangement being such as to permit continuous withdrawal of silica as ingot at a rate substantially similar to that at which silica is deposited by the synthesis burner(s).

2. A furnace according to claim 1, wherein the moveable support means comprises an arrangement of moveable clamps.

3. A furnace according to claim 2, wherein the refractory container with its die, the ingot and the arrangement of clamps can be rotated synchronously to provide a deposited glass of improved homogeneity.

4. A furnace according to claim 2 or claim 3, wherein the refractory container with its die, the ingot and the arrangement of clamps can be moved to and fro horizontally to permit spreading of the pattern of deposited glass from the burner(s).

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5. A furnace according to claim 2 or claim 3 wherein the refractory container with its die, the ingot and the arrangement of clamps can be moved in orthogonally disposed x- and y- directions, to permit spreading of the pattern of deposited glass from the one or more burners.

6. A furnace according to claim 2 or claim 3, wherein spreading of the pattern of deposited silica is achieved by movement of the burner or burner array and/or of the refractory container.

7. A method of forming a shaped body of synthetic vitreous silica including the steps of: generating a melt of synthetic vitreous silica contained in a refractory container, part of the boundary of which defines a shaping orifice; maintaining the melt in a molten state by heating with one or more burners; and removing the generated synthetic vitreous silica through the orifice as a shaped ingot; characterised in that at least one burner is a synthesis burner, and the silica is deposited from such synthesis burner(s) in such a manner that synthetic vitreous silica can be deposited at a rate substantially similar to that at which silica is withdrawn as ingot through the shaping orifice.

8. A method according to claim 7, wherein the shaping orifice is located at the lowest part of the mass in the refractory container and the removal involves positively withdrawing the ingot from below.

9. A method according to claim 7 or claim 8, wherein the synthesis burner(s) serves to heat the surface of the melt so that the deposited silica sinters directly to glass.

AMENDED SHEET

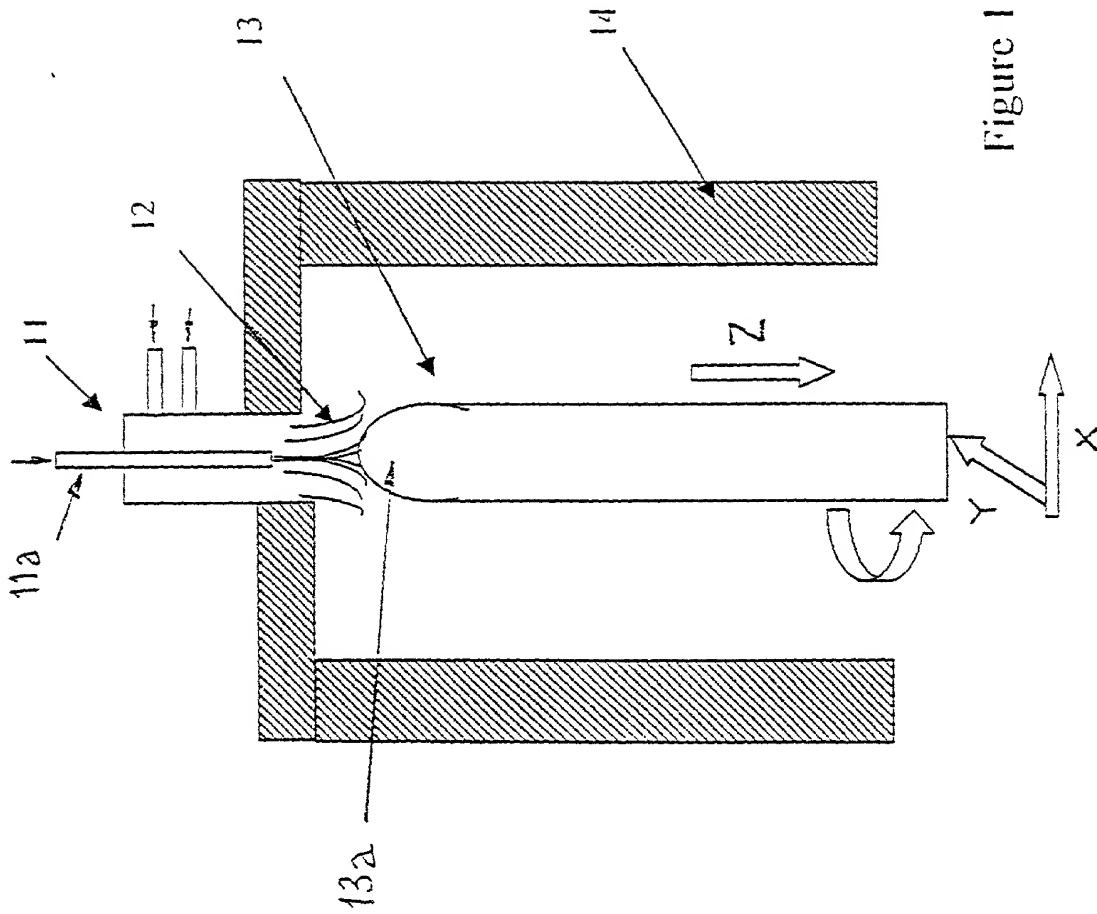


Figure 1

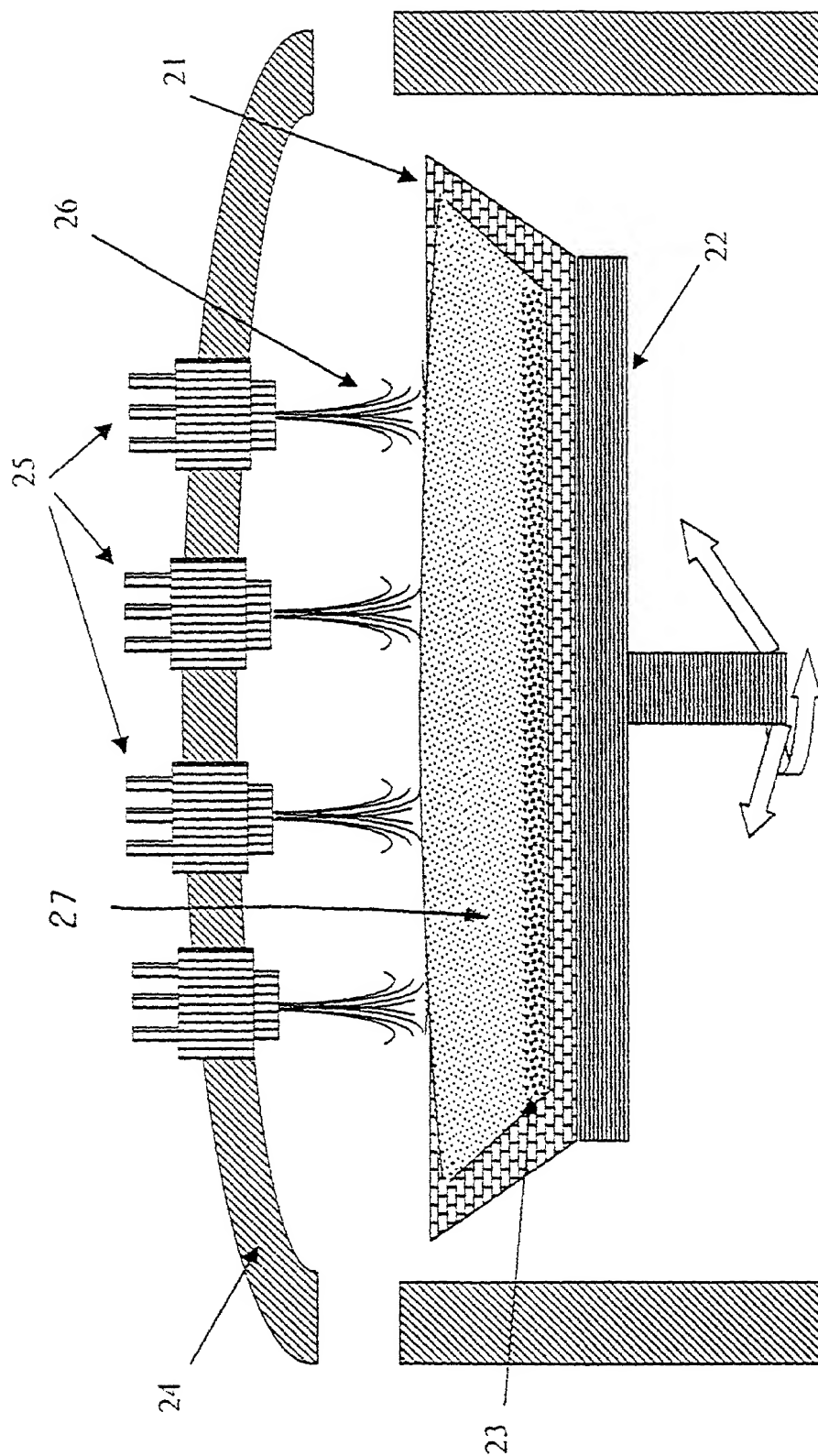


Figure 2

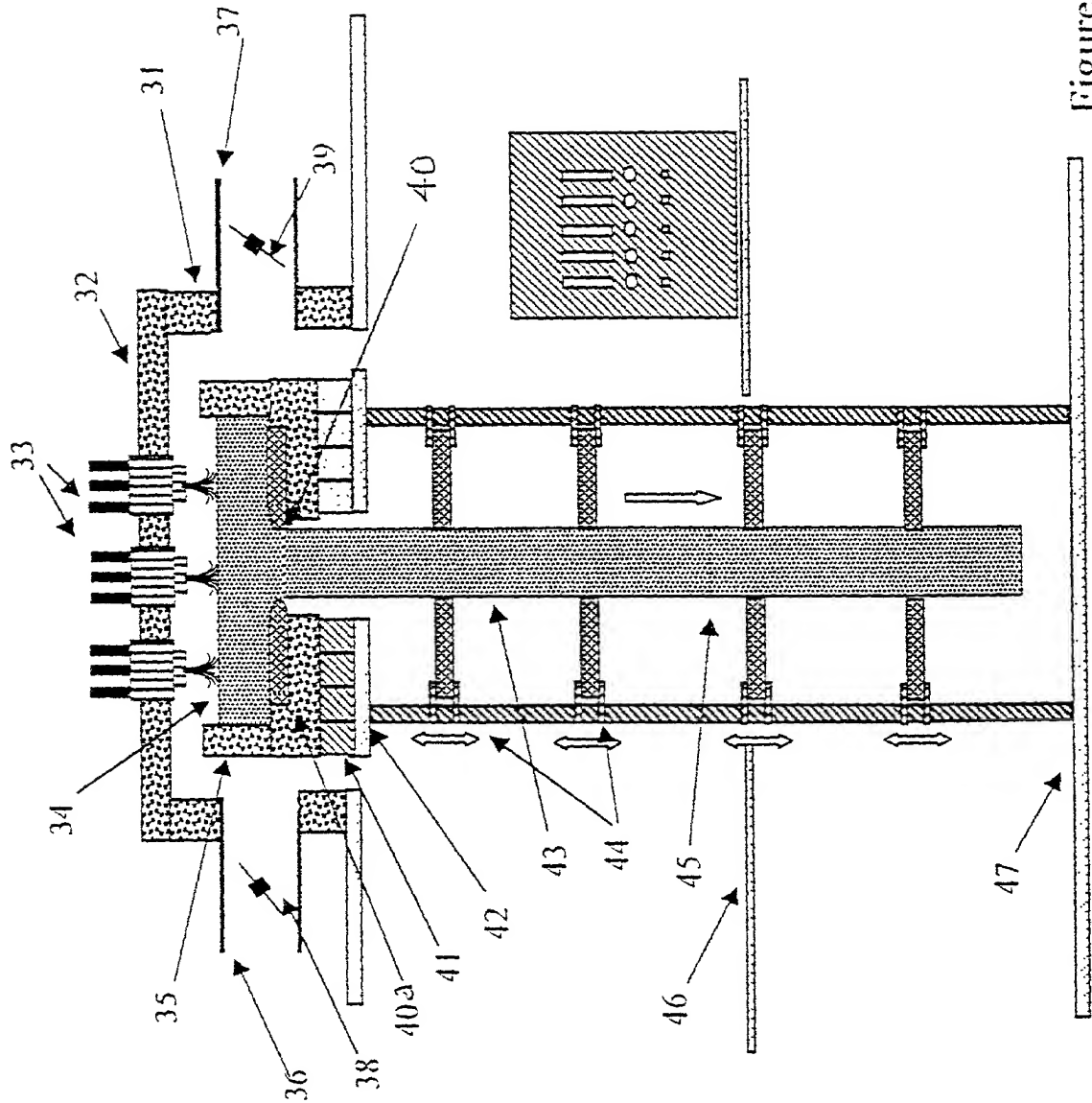


Figure 3

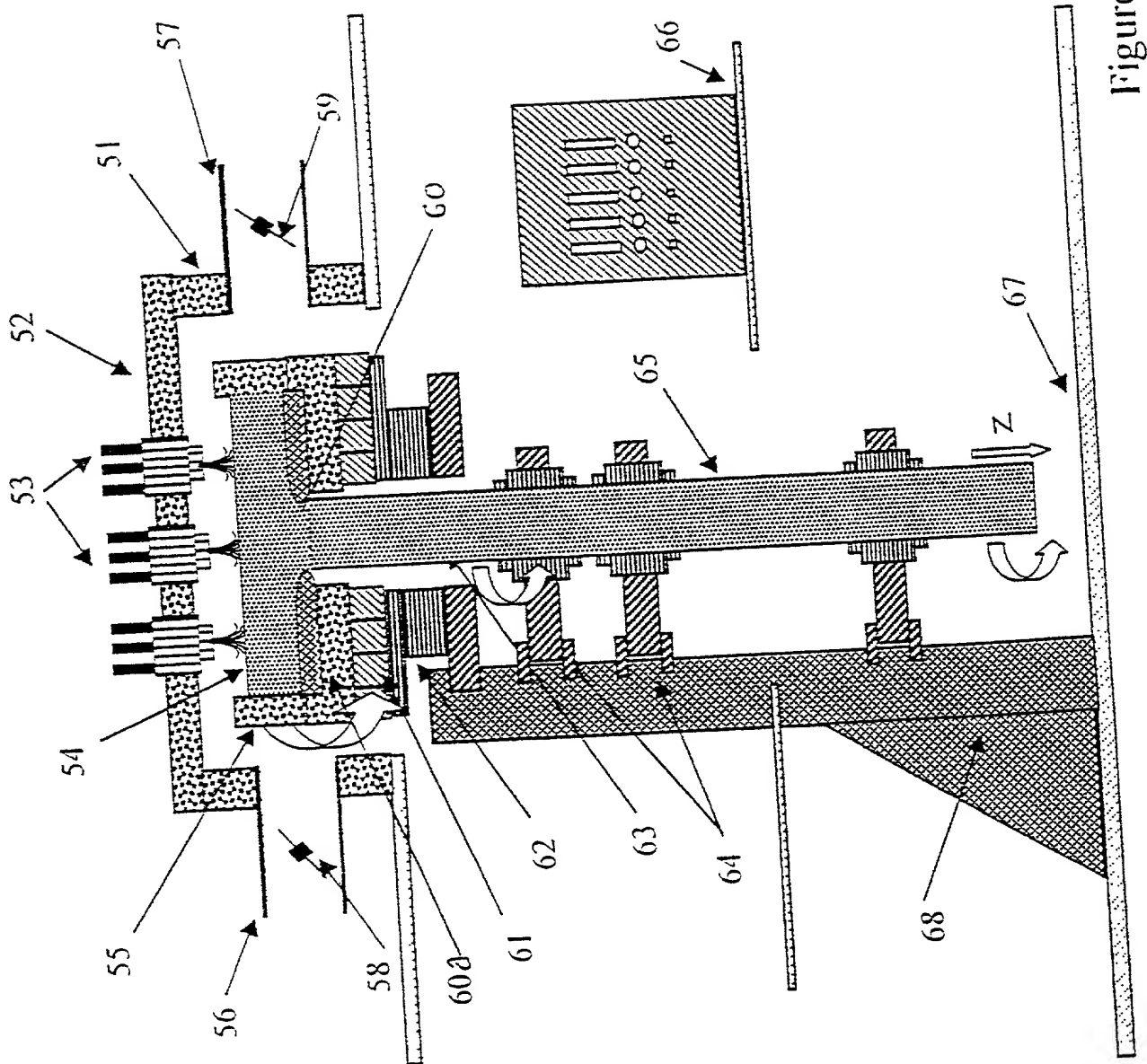


Figure 4

COMBINED DECLARATION AND POWER OF ATTORNEY(ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL, DIVISIONAL,
CONTINUATION, OR C-I-P)

As a below named inventor, I hereby declare that:

TYPE OF DECLARATION

This declaration is of the following type:

(check one applicable item below)

- ☐ original.
☐ design.
☐ supplemental.

NOTE: If the declaration is for an International Application being filed as a divisional, continuation or continuation-in-part application, do not check next item; check appropriate one of last three items.

- ☒ national stage of PCT.

NOTE: If one of the following 3 items apply, then complete and also attach ADDED PAGES FOR DIVISIONAL, CONTINUATION OR C-I-P.

NOTE: See 37 C.F.R. § 1.63(d) (continued prosecution application) for use of a prior nonprovisional application declaration in the continuation or divisional application being filed on behalf of the same or fewer of the inventors named in the prior application.

- ☐ divisional.
☐ continuation.

NOTE: Where an application discloses and claims subject matter not disclosed in the prior application, or a continuation or divisional application names an inventor not named in the prior application, a continuation-in-part application must be filed under 37 C.F.R. § 1.53(b) (application filing requirements — nonprovisional application).

- ☐ continuation-in-part (C-I-P).

INVENTORSHIP IDENTIFICATION

WARNING: If the inventors are each not the inventors of all the claims, an explanation of the facts, including the ownership of all the claims at the time the last claimed invention was made, should be submitted.

My residence, post office address and citizenship are as stated below, next to my name. I believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter that is claimed, and for which a patent is sought on the invention entitled:

TITLE OF INVENTION

Process and apparatus for manufacturing a glass
ingot from synthetic silica

SPECIFICATION IDENTIFICATION

the specification of which:

(complete (a), (b), or (c))

(a) ☐ is attached hereto.

NOTE: "The following combinations of information supplied in an oath or declaration filed on the application filing date with a specification are acceptable as minimums for identifying a specification and compliance with any one of the items below will be accepted as complying with the identification requirement of 37 CFR 1.63:

"(1) name of inventor(s), and reference to an attached specification which is both attached to the oath or declaration at the time of execution and submitted with the oath or declaration on filing;

"(2) name of inventor(s), and attorney docket number which was on the specification as filed; or

"(3) name of inventor(s), and title which was on the specification as filed."

Notice of July 13, 1995 (1177 O.G. 60).

(b) ☐ was filed on _____, as ☐ Serial No. 0 / _____
or ☐ _____
and was amended on _____ (if applicable).

NOTE: Amendments filed after the original papers are deposited with the PTO that contain new matter are not accorded a filing date by being referred to in the declaration. Accordingly, the amendments involved are those filed with the application papers or, in the case of a supplemental declaration, are those amendments claiming matter not encompassed in the original statement of invention or claims. See 37 CFR 1.67.

NOTE: "The following combinations of information supplied in an oath or declaration filed after the filing date are acceptable as minimums for identifying a specification and compliance with any one of the items below will be accepted as complying with the identification requirement of 37 CFR 1.63:

"(1) name of inventor(s), and application number (consisting of the series code and the serial number; e.g., 08/123,456);

"(2) name of inventor(s), serial number and filing date;

"(3) name of inventor(s) and attorney docket number which was on the specification as filed;

"(4) name of inventor(s), title which was on the specification as filed and filing date;

"(5) name of inventor(s), title which was on the specification as filed and reference to an attached specification which is both attached to the oath or declaration at the time of execution and submitted with the oath or declaration; or

"(6) name of inventor(s), title which was on the specification as filed and accompanied by a cover letter accurately identifying the application for which it was intended by either the application number (consisting of the series code and the serial number; e.g., 08/123,456), or serial number and filing date. Absent any statement(s) to the contrary, it will be presumed that the application filed in the PTO is the application which the inventor(s) executed by signing the oath or declaration."

Notice of July 13, 1995 (1177 O.G. 60), M.P.E.P. § 601.01(a), 6th ed., rev. 3.

(c) ☒ was described and claimed in PCT International Application No. PCT/GB99/02278, filed on July 15, 1999 and as amended under PCT Article 19 on _____ (if any).

SUPPLEMENTAL DECLARATION (37 C.F.R. § 1.67(b))

(complete the following where a supplemental declaration is being submitted)

- ☐ I hereby declare that the subject matter of the
- ☐ attached amendment
 - ☐ amendment filed on _____

was part of my/our invention and was invented before the filing date of the original application, above-identified, for such invention.

ACKNOWLEDGEMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information, which is material to patentability as defined in 37, Code of Federal Regulations, § 1.56,

(also check the following items, if desired)

- ☐ and which is material to the examination of this application, namely, information where there is a substantial likelihood that a reasonable Examiner would consider it important in deciding whether to allow the application to issue as a patent, and
- ☐ in compliance with this duty, there is attached an information disclosure statement, in accordance with 37 CFR 1.98.

PRIORITY CLAIM (35 U.S.C. §§ 119(a)-(d))

NOTE: "The claim to priority need be in no special form and may be made by the attorney or agent if the foreign application is referred to in the oath or declaration as required by § 1.63. The claim for priority and the certified copy of the foreign application specified in 35 U.S.C. 119(b) must be filed in the case of an interference (§ 1.630), when necessary to overcome the date of a reference relied upon by the examiner, when specifically required by the examiner, and in all other situations, before the patent is granted. If the claim for priority or the certified copy of the foreign application is filed after the date the issue fee is paid, it must be accompanied by a petition requesting entry and by the fee set forth in § 1.17(i). If the certified copy is not in the English language, a translation need not be filed except in the case of interference; or when necessary to overcome the date of a reference relied upon by the examiner; or when specifically required by the examiner, in which event an English language translation must be filed together with a statement that the translation of the certified copy is accurate." 37 C.F.R. § 1.55(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §§ 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

(complete (d) or (e))

- (d) ☐ no such applications have been filed.
- (e) ☒ such applications have been filed as follows.

NOTE: Where item (c) is entered above and the International Application which designated the U.S. itself claimed priority check item (e), enter the details below and make the priority claim.

(Declaration and Power of Attorney [1-1]—page 3 of 7)

**PRIOR FOREIGN/PCT APPLICATION(S) FILED WITHIN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS APPLICATION
AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. § 119(a)-(d)**

COUNTRY (OR INDICATE IF PCT)	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 37 USC 119
GB	9815357.0	15 July 1998	<input checked="" type="checkbox"/> YES NO <input type="checkbox"/>
			<input type="checkbox"/> YES NO <input type="checkbox"/>
			<input type="checkbox"/> YES NO <input type="checkbox"/>
			<input type="checkbox"/> YES NO <input type="checkbox"/>
			<input type="checkbox"/> YES NO <input type="checkbox"/>

**CLAIM FOR BENEFIT OF PRIOR U.S. PROVISIONAL APPLICATION(S)
(34 U.S.C. § 119(e))**

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below:

PROVISIONAL APPLICATION NUMBER

FILING DATE

_____ / _____	_____
_____ / _____	_____
_____ / _____	_____

**CLAIM FOR BENEFIT OF EARLIER US/PCT APPLICATION(S)
UNDER 35 U.S.C. 120**

- ☐ The claim for the benefit of any such applications are set forth in the attached ADDED PAGES TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR DIVISIONAL, CONTINUATION OR CONTINUATION-IN-PART (C-I-P) APPLICATION.

**ALL FOREIGN APPLICATION(S), IF ANY, FILED MORE THAN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS U.S. APPLICATION**

NOTE: If the application filed more than 12 months from the filing date of this application is a PCT filing forming the basis for this application entering the United States as (1) the national stage, or (2) a continuation, divisional, or continuation-in-part, then also complete ADDED PAGES TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR DIVISIONAL, CONTINUATION OR C-I-P APPLICATION for benefit of the prior U.S. or PCT application(s) under 35 U.S.C. § 120.

POWER OF ATTORNEY

I hereby appoint the following practitioner(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

(list name and registration number)

Stanley B. Kita, Registration No. 24,561; George A. SMITH, Jr.,
Registration No. 24,442; Wilson OBERDORFER, Registration
No. 17,379; Mary E. BAK, Registration No. 31,215; Henry HANSEN,
Registration No. 19,612 and Cathy Ann KODROFF, Registration No. 33,980
(check the following item, if applicable)

- ☐ I hereby appoint the practitioner(s) associated with the Customer Number provided below to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.
- ☒ Attached, as part of this declaration and power of attorney, is the authorization of the above-named practitioner(s) to accept and follow instructions from my representative(s).

SEND CORRESPONDENCE TO

DIRECT TELEPHONE CALLS TO:
(Name and telephone number)

☐ Address

~~HOWSON AND HOWSON~~
~~Spring House, Corporate Center,~~
~~P.O. Box 457, Spring House,~~
~~Pennsylvania 19477~~

(215) 540-9200

☐ Customer Number _____

DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE(S)

NOTE: Carefully indicate the family (or last) name, as it should appear on the filing receipt and all other documents.

Full name of sole or first inventor

IAN GEORGE SAYCE
(GIVEN NAME) (MIDDLE INITIAL OR NAME) FAMILY (OR LAST NAME)

Inventor's signature [Signature]

Date January 15, 2001 Country of Citizenship United Kingdom

Residence United Kingdom

Post Office Address 21 Crabtree Road, Stocksfield, Northumberland
NE43 7NX, United Kingdom [Signature]

Full name of second joint inventor, if any

PETER JOHN WELLS
(GIVEN NAME) (MIDDLE INITIAL OR NAME) FAMILY (OR LAST NAME)

Inventor's signature [Signature]

Date 15 January 2001 Country of Citizenship United Kingdom

Residence United Kingdom

Post Office Address 2 Linden Way, Gateshead, Tyne and Wear
NE9 7BL, United Kingdom [Signature]

Full name of third joint inventor, if any

(GIVEN NAME) (MIDDLE INITIAL OR NAME) FAMILY (OR LAST NAME)

Inventor's signature _____

Date _____ Country of Citizenship _____

Residence _____

Post Office Address _____

(check proper box(es) for any of the following added page(s)
that form a part of this declaration)

- ☐ **Signature** for fourth and subsequent joint inventors. *Number of pages added* _____

* * *

- ☐ **Signature** by administrator(trix), executor(trix) or legal representative for deceased or incapacitated inventor. *Number of pages added* _____

* * *

- ☐ **Signature** for inventor who refuses to sign or cannot be reached by person authorized under 37 CFR 1.47. *Number of pages added* _____

* * *

- ☐ **Added page for signature** by one joint inventor on behalf of deceased inventor(s) where legal representative cannot be appointed in time. (37 CFR 1.47)

* * *

- ☐ **Added pages to combined declaration and power of attorney for divisional, continuation, or continuation-in-part (C-I-P) application.**

☐ **Number of pages added** _____

* * *

- ☒ **Authorization of practitioner(s) to accept and follow instructions from representative.**

* * *

(if no further pages form a part of this Declaration,
then end this Declaration with this page and check the following item)

- ☐ **This declaration ends with this page.**

ADDED PAGE TO COMBINED DECLARATION AND POWER OF ATTORNEY
FOR AUTHORIZATION OF ATTORNEY(S) TO ACCEPT AND FOLLOW
INSTRUCTIONS FROM REPRESENTATIVE

The undersigned to this declaration and power of attorney hereby authorizes the U.S. attorney(s) named herein to accept and follow instructions from

JY & GW Johnson

Name(s) of authorized representative(s)

Kingsbourne House,

Address

229-231 High Holborn,

London WC1V 7DP ENGLAND

as to any actions to be taken in the Patent and Trademark Office regarding this application without direct communication between the U.S. attorney(s) and the undersigned. In the event of a change in the person(s) from whom instructions may be taken, the U.S. attorney(s) will be so notified by the undersigned.

(Added page to Combined Declaration and Power of Attorney for authorization of attorney(s) to accept and follow instructions from representative [1-24])